SECTION 2 TECHNICAL PART

2.4 Confirmation analysis by GC/C/IRMS

2.4.1 GC/MS analysis

TOPLEVEL PARAMETERS

```
_lethod Information For: C:\MSDCHEM\1\METHODS\MAN_52.M
ethod Sections To Run:
  ( ) Save Copy of Method With Data
                         Pre-Run Cmd/Macro =
_ ( ) MSTOP
  ( ) Instrument Control Pre-Run Cmd/Macro =
  ( ) Data Analysis Pre-Run Cmd/Macro =
  (X) Data Acquisition
_ (X) Data Analysis
                         Post-Run Cmd/Macro =
 ( ) MSTOP
( ) Instrument Control Post-Run Cmd/Macro = ( ) Data Analysis Post-Run Cmd/Macro =
1ethod Comments:
  This is the default method
                               END OF TOPLEVEL PARAMETERS
                                INSTRUMENT CONTROL PARAMETERS
                                OVEN
                                             Maximum temp: 325 'C
   Initial temp: 70 'C (On)
Initial time: 1.00 min
                                             Equilibration time: 0.50 min
      # Rate Final temp Final time
1 30.00 270 12.00
2 10.00 300 3.00
    Ramps:
      3 0.0(Off)
    Post temp: 70 'C
Post time: 0.00 min
Run time: 25.67 min
                                     BACK INLET (UNKNOWN)
 FRONT INLET (SPLIT/SPLITLESS)
    Mode: Splitless
    Initial temp: 280 'C (On)
    Pressure: 145.0 kPa (On)
Purge flow: 20.0 mL/min
Purge time: 2.00 min
Total flow: 25.4 mL/min
Gas saver: Off
Gas type: Helium
                                           COLUMN 2
  FOLUMN 1
                                               (not installed)
   Capillary Column
    Model Number: Agilent 19091s-433
Max temperature: 325 °C
Nominal length: 30.0 m
     Nominal diameter: 250.00 um
     Nominal film thickness: 0.25 um
     Mode: constant pressure
    Pressure: 145.0 kPa
     Nominal initial flow: 2.3 mL/min
     Average velocity: 56 cm/sec
     Inlet: Front Inlet
     Outlet: MSD
     Outlet pressure: vacuum
                                                                       Page: 1
                               Mon Jul 24 14:19:20 2006
  Method: MAN_52.M
```

```
BACK DETECTOR ()
. RONT DETECTOR ()
                                              SIGNAL 2
:IGNAL 1
                                                 Data rate: 20 Hz
Data rate: 20 Hz
Type: test plot
                                                 Type: test plot
                                                 Save Data: Off
Save Data: Off
                                                 Zero: 0.0 (Off)
  Zero: 0.0 (Off)
Range: 0
                                                 Range: 0
  Fast Peaks: Off
Attenuation: 0
                                                Fast Peaks: Off
                                                 Attenuation: 0
                                              COLUMN COMP 2
COLUMN COMP 1
                                                 (No Detectors Installed)
  (No Detectors Installed)
THERMAL AUX 2
   Use: MSD Transfer Line Heater
   Description:
  Initial temp: 300 'C (On)
Initial time: 0.00 min
      # Rate Final temp Final time
1 0.0(Off)
                                            POST RUN
                                                 Post Time: 0.00 min
IME TABLE
                                                  Parameter & Setpoint
               Specifier
    Time
                               7673 Injector
      Front Injector:
                                            0
       Sample Washes
         Sample Pumps
                                         1.0 microliters
         Injection Volume
                                       10.0 microliters
     Syringe Size

PostInj Solvent A Washes

PostInj Solvent B Washes

Viscosity Delay

Plunger Speed

PreInjection Dwell

PostInjection Dwell

PostInjection Dwell

10.0 microlit

3

Seconds

Fast

0.00 minutes
         Syringe Size
     Back Injector:
 o parameters specified
  Column 1 Inventory Number : ?
 Column 2 Inventory Number :
                                     MS ACQUISITION PARAMETERS
 General Information
 -----
                             : atune.u
: Scan
_Tune File
Acquistion Mode
 MS Information
|-----
                             ; 9,00 min
 Solvent Delay
                             : False
EM Absolute
                            ·: 0
  EM Offset
Resulting EM Voltage : 1811.8
[ | Scan Parameters]
                             : 50.0
Low Mass
                                                                              Page: 2
                                 Mon Jul 24 14:19:20 2006
' Method: MAN 52.M
```

```
: 550.0
: 150
igh Mass
hreshold
                                  A/D Samples 4
                     : 2
: 50.0
: 550.0
Sample # Plot 2 low mass
lot 2 high mass
[MSZones]
                        : 150 C maximum 200 C
S Quad
                        : 230 C maximum 250 C
S Source
                            END OF MS ACQUISITION PARAMETERS
                          END OF INSTRUMENT CONTROL PARAMETERS
                           DATA ANALYSIS PARAMETERS
Method Name: C:\MSDCHEM\1\METHODS\MAN_52.M
Percent Report Settings
1 |-----
Gort By: Signal
Tutput Destination
   Screen: No
    Printer: Yes
            No
    File:
Integration Events: AutoIntegrate
 Generate Report During Run Method: No
 ignal Correlation Window: 0.020
' ualitative Report Settings
 [ ------
 Peak Location of Unknown: Apex
 Library to Search Minimum Quality C:\temp\IRMS.L 90
Integration Events: AutoIntegrate
 Report Type: Summary
 Dutput Destination
  Screen: No
Printer: Yes
    File:
             No
Senerate Report During Run Method: No
  Quantitative Report Settings
                   Mon Jul 24 14:19:20 2006
                                                                   Page: 3
Method: MAN_52.M
```

_aport Type: Summary putput Destination Screen: Yes Printer: No File: No enerate Report During Run Method: No alibration Last Updated: pference Window: 10.00 Percent on-Reference Window: 5.00 Percent Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Efault Sample Concentration: 0.00 Compound Information *** Empty Quantitation Database *** END OF DATA ANALYSIS PARAMETERS Mon Jul 24 14:19:20 2006

Method: MAN_52.M

Mon Jul 24 14:19:20 2006

Page: 4

```
dditional Information for STER1FS.M
ile created Fri Mar 05 17:08:58 2004
```

ri Mar 05 17:08:58 2004

ethod : C:\MSDCHEM\1\METHODS\STER1FS.M enamed: C:\MSDCHEM\1\METHODS\STER1FSKETO.M

tue Apr 20 14:52:43 2004

ethod : C:\MSDCHEM\1\METHODS\STER1FS.M denamed: C:\MSDCHEM\1\METHODS\MAN_52.M ved Nov 09 18:43:55 2005

Method: MAN_52.M

Mon Jul 24 14:19:20 2006

Page: 5

```
Sequence Name: C:\MSDChem\1\sequence\2006\Jui106\2307.S
         Comment:
        Operator: 49
       Data Path: D:\MSD22\JUIL06\2307\
Top
                    Pre-Seq Cmd:
Instrument Control Pre-Seq Cmd:
                   Pre-Seq Cmd:
 Data Analysis
Top
                   Post-Seq Cmd:
Instrument Control Post-Seq Cmd:
Data Analysis Post-Seq Cmd:
    Method Sections To Run On A Barcode Mismatch
(X) Full Method (X) Inject Anyway
                                ( ) Don't Inject
     ( ) Reprocessing Only
                      Sample Name/Misc Info
   Line

    Calibration

                      2307MixAc01
      Datafile
                      MAN_52
      Method
                   2 blu1F3
                               MAN_52 Blu 1 F3
  2)
     Blank
⇒ 3) Sample
                   3
     Datafile
                      17807474F3
                      MAN_52
     Method
                   4 blulF1
                               MAN_52 Blu 1 F1
 4) Blank
5) Sample
                   5
                      17807474F1
      Datafile
                      MAN_52
      Method
                   6 blulF2
  6) Blank
                               MAN_52 Blu 1 F2
5 7) Sample
                      17807474F2
      Datafile
      Method
                      MAN 52
  8) Sample
                      17807474F2b
      Datafile
      Method
                      MAN_52
```

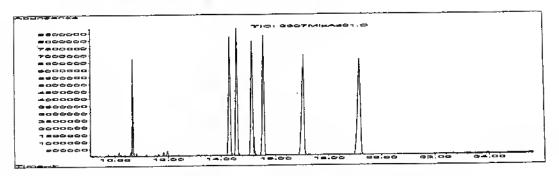
Séquence vérifiée	par :	9
Remarques:		min Maria
*************************	- 100	14

Last Modified: Sun Jul 23 10:17:10 2006

Page: 1

D:\Msd22\Juil06\2307\2307MixAc01.D

Data File Name 2307MixAc01.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 10:19
Acq. Method File MAN_52.M
Sample Name Mix Ac 50
Vial Number 1
Misc Info Mix Acétate 001 50 ng injecté



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstenol AC	10.69		258	10,892,705
Etiocholanolone AC	14.35	1.343	272	20,821,485
Androsterone AC	14.62	1.367	272	35, S 20,111
5b Androstan 3a 17b diol diAC	15.17	1.419	256	20,304,691
5e Androsten 3a 17b diol diAC	15.57	1.456	316	28,264,859
11 KetoEtiocholenolone AC	17.07	1.597	271	27,735,914
5b Pregnan 3a 20a diol diAC	19.20	1,796	284	35,625,886

M2 signal

Q1 signal	Q1 Response	Q1 Ratio
243	11,496,221	105.5
257	13,275,551	63.8
257	15,144,189	42.6
316	18,349,659	90.4
	16,541,690	58.5
	•	83.0
	, ,	56.3
	257	243 11,496,221 257 13,275,551 257 15,144,189 316 18,349,659 241 16,541,690 191 23,029,104

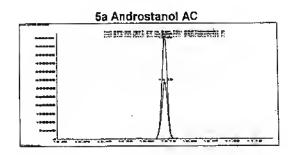
M3 signal

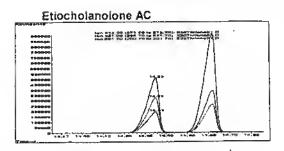
Page 1 of 2

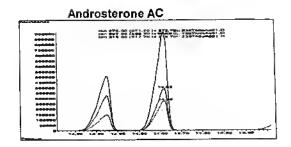
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	6,139,162	56.4
Etiocholanolone AC	201	7,984,811	38.3
Androsterone AC	218	10,868,162	30.6
	241	15,646,481	77.1
5b Androstan 3a 17b diol diAC	256	13,817,358	48.9
5a Androstan 3a 17b diol diAC	286	16,604,214	59.9
11 KetoEtiocholanolone AC		9,962,084	28.0
5b Pregnan 3a 20a diol diAC	344	9,902,004	20,0

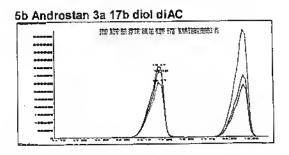
C:\MSDCHEM\CUSTRPT\MAN52.CRT

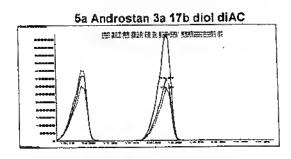
7/23/2006 11:33 AM

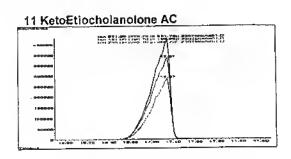


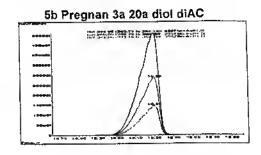












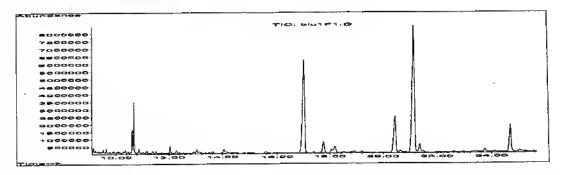
Page 2 of 2

C:\MSDCHEM\CUSTRPT\MAN52.CRT

7/23/2006 11:33 AM

D:\Msd22\Juil06\2307\biu1F1.D

Data File Name blu1F1.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 12:10
Acq. Method File MAN_52.M
Sample Name Blu 1 F1
Vial Number 4
Misc Info Blanc urinaire 1 Pool 4 Fraction 1 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanoi AC	10.68		258	5,234,507
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	0.00	. 0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	17.04	1.596	271	23,056,534
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

M2 signal

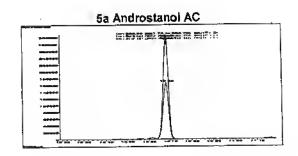
Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	5,447,955	104.1
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	19,045,346	82.6
5b Pregnan 3a 20a diol diAC	269		0.0

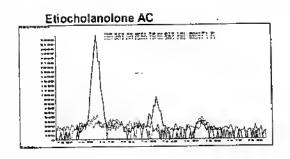
M3 signal

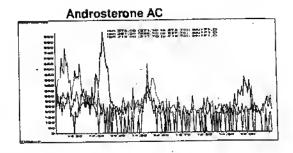
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	2,943,791	56.2
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	13,914,444	60.3
5b Pregnan 3a 20a diol diAC	344	0	0.0

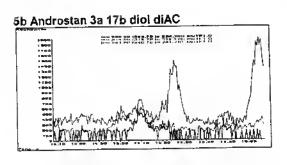
C:\MSDCHEM\CUSTRPT\MAN52.CRT

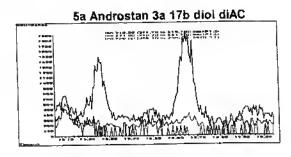
7/23/2006 12:55 PM

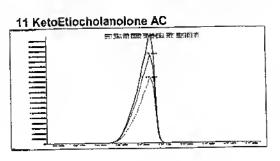


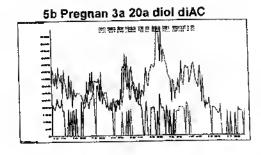












Page 2 of 2

C:\MSDCHEM\CUSTRPT\MAN52.CRT

7/23/2006 12:55 PM

D:\Msd22\Juli06\2307\17807474F1.D

Data File Name 17807474F1.D

Data File Path D:\Msd22\Juil06\2307\

Operator 49

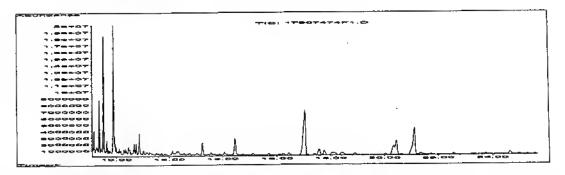
Date Acquired 7/23/2006 12:42

Acq. Method File MAN_52.M

Sample Name 178/07 995474 F1

Vial Number 5

Misc Info 178/07 995474 Fraction 1 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Ret Time	Rel Ret Time	Target Signal	Target Response
10.69		258	2,672,512
0.00	0.000	272	0
0.00	0.000	272	0
0.00	0.000	256	0
0.00	0.000	316	0
****	1.596	271	27,520,882
		284	0
	10.69 0.00 0.00	10.69 0.00 0.000 0.00 0.000 0.00 0.000 0.00 0.000 17.05 1.596	10.69 258 0.00 0.000 0.00 0.000 0.00 0.000 0.00 0.000 0.00 0.000 316 17.05 1.596 271

M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	2,977,015	111.4
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	22,323,512	81.1
5b Pregnan 3a 20a diol diAC	269	0	0.0

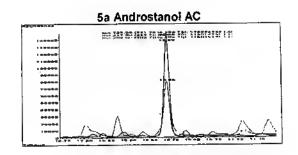
M3 signal

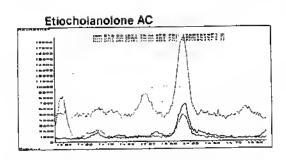
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	1,719,112	64.3
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	16,694,242	60.7
5b Pregnan 3a 20a diol diAC	344	0	0.0

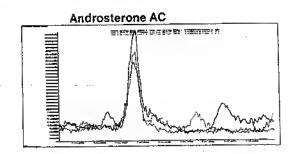
Page 1 of 2

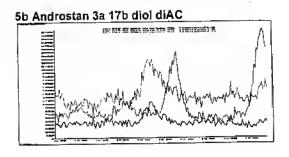
C:\MSDCHEM\CUSTRPT\MAN52.CRT

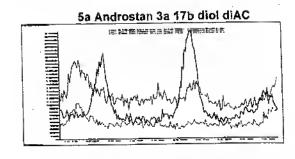
7/23/2006 1:14 PM

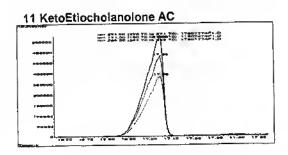


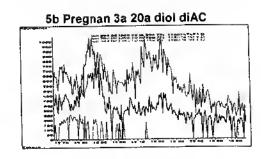












C:\MSDCHEM\CUSTRPT\MAN52.CRT

7/23/2006 1:14 PM

D:\Msd22\Juil06\2307\blu1F2.D

Data File Name blu1F2.D

Data File Path D:\Msd22\Juil06\2307\

Operator 49

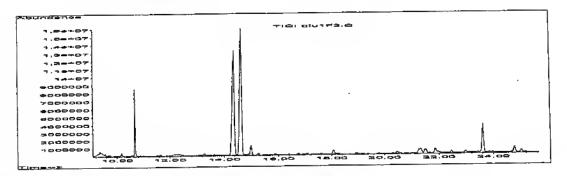
Date Acquired 7/23/2006 13:15

Acq. Method File MAN_52.M

Sample Name Blu 1 F2

Vial Number 6

Misc Info Blanc urinaire 1 Pool 4 Fraction 2 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Ref Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	14,356,335
Etiocholanolone AC	14.39	1.347	272	46,218,242
	14.66	1.373	272	92,159,652
Androsterone AC	0.00	0.000	256	0
5b Androstan 3a 17b diol diAC		0.000	316	n
5a Androstan 3a 17b diol diAC	0.00			n
11 KetoEtiocholanolone AC	0.00	0,000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	U

M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	14,913,181	103.9
Etiocholanolone AC	257	29,244,244	63.3
Androsterone AC	257	39,385,747	42.7
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
	191	0	0.0
11 KetoEtiocholanolone AC	269	ñ	0.0
5b Pregnan 3a 20a diol diAC	209		Y.V

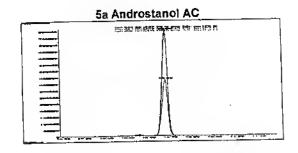
M3 signal

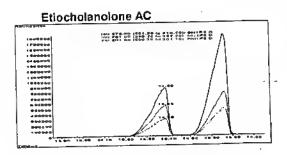
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	7,892,527	55.0
Etiocholanolone AC	201	16,995,418	36.8
Androsterone AC	218	26,814,361	29.1
5b Androstan 3a 17b diol diAC	241	0	0.0
	256	n	0.0
5a Androstan 3a 17b diol diAC	286	Ď	0.0
11 KetoEtiocholanolone AC		0	0.0
5b Pregnan 3a 20a diol diAC	344		

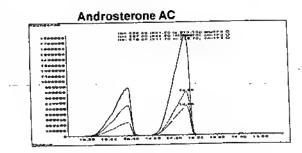
C:\MSDCHEM\CUSTRPT\MAN52.CRT

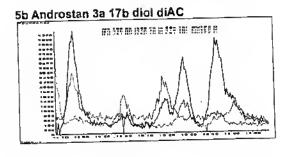
7/23/2006 2:23 PM

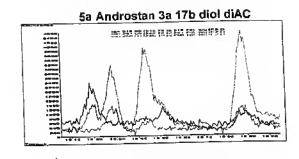
Page 1 of 2

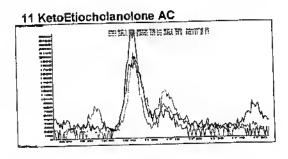


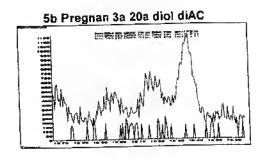












C:\M5DCHEM\CU5TRPT\MAN52.CRT

7/23/2006 2:**2**3 PM

D:\Msd22\Juii06\2307\17807474F2b.D

Data File Name 17807474F2b.D

Data File Path D:\Msd22\Juil06\2307\

Operator 49

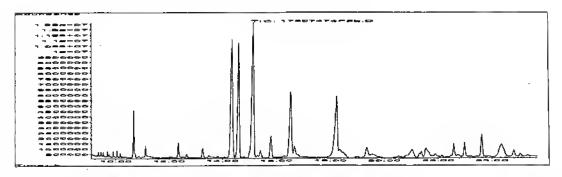
Date Acquired 7/23/2006 14:33

Acq. Method File MAN_52.M

Sample Name 178/07 995474 F2

Vial Number 7

Misc Info 178/07 995474 Fraction 2 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	6,790,329
Etiocholanolone AC	14.38	1.345	272	30,616,404
Androsterone AC	14.65	1.370	272	44,803,237
5b Androstan 3a 17b dioi diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0 .
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	0

M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	7,142,048	105.2
Etiocholanolone AC	257	19,396,055	63.4
Androsterone AC	257	19,255,218	43.0
5b Androstan 3a 17b diol diAC	316	0	0.0
5a Androstan 3a 17b diol diAC	241	0	0.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	. 0	0.0

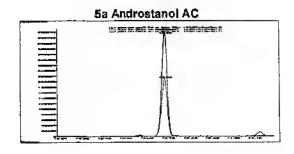
M3 signal

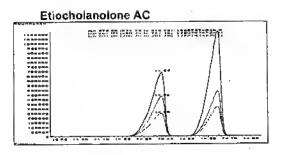
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	3,868,152	57.0
Etiocholanolone AC	201	11,568,610	37.8
Androsterone AC	218	13,516,338	30.2
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0	0.0

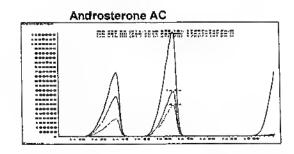
Page 1 of 2

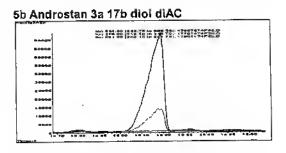
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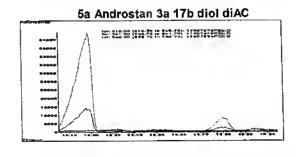
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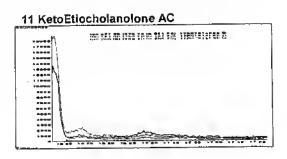


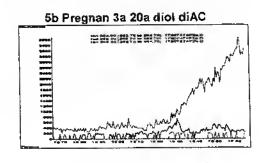












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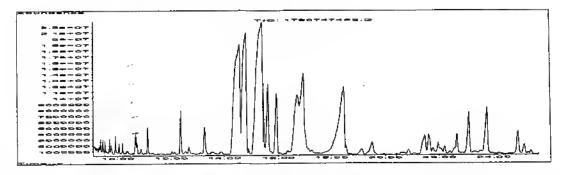
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Data File Name 17807474F2.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 13:47
Acq. Method File MAN_52.M
Sample Name 178/07 995474 F2

Vial Number 7

Misc Info 178/07 995474 Fraction 2 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.69		258	4,709,923
Etiocholanolone AC	14.56	1.365	272	169,848,822
Androsterone AC	14.84	1.388	272	213,976,918
5b Androstan 3a 17b diol diAC	0.00	0.000	256	0
5a Androstan 3a 17b diol diAC	0.00	0.000	316	0
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	0.00	0.000	284	00

M2 signal

1.

Q1 signal	Q1 Response	Q1 Ratio
243	5,011,709	106.4
257	104,358,276	61.4
257	91,230,541	42.6
316	0 -	0.0
241	0	0.0
191	0	0.0
269	0	0.0
	243 257 257 316 241 191	243 5,011,709 257 104,356,276 257 91,230,541 316 0 241 0 191 0

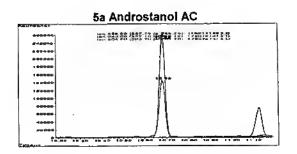
M3 signal

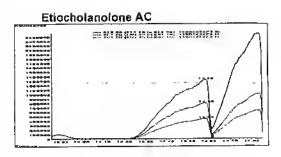
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstanol AC	204	2,896,716	61.5
Etiocholanolone AC	201	59,514,197	35.0
Androsterone AC	218	61,028,102	28.5
5b Androstan 3a 17b diol diAC	241	0	0.0
5a Androstan 3a 17b.diol diAC	256	0	0.0
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	0	0.0

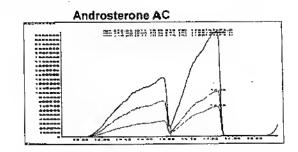
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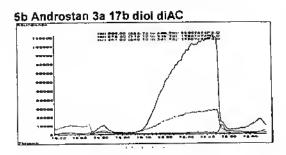
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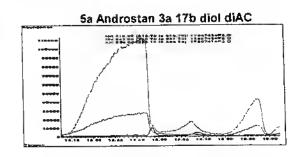
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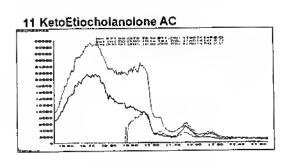


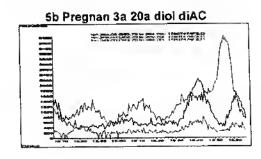












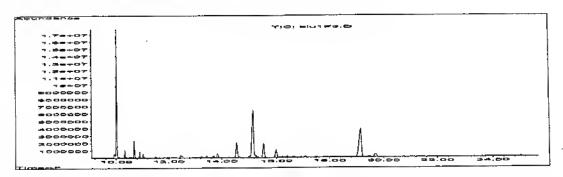
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Data File Name blu1F3.D
Data File Path D:\Msd22\Juil06\2307\
Operator 49
Date Acquired 7/23/2006 11:00
Acq. Method File MAN_52.M
Sample Name Blu 1 F3
Vial Number 2

Misc Info Blanc urinaire 1 Pool 4 Fraction 3 dans 100µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.68		258	3,476,361
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	15.17	1.420	256	15,597,241
5a Androstan 3a 17b diol diAC	15.51	1.452	316	5,215,670
11 KetoEtiocholanolone AC	0.00	0.000	271	0
5b Pregnan 3a 20a diol diAC	19.14	1.792	284	16,549,438

M2 signal

Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	3,650,999	105.0
Etiocholanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	14,170,551	90.9
5a Androstan 3a 17b diol diAC	241	3,187,095	61.1
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	9.365.521	56.6

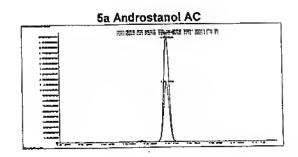
M3 signal

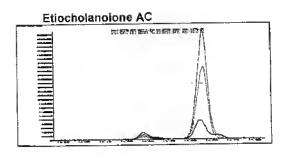
Name	Q2 signal	Q2 Response	Q2 Ratio
5a Androstenol AC	204	1,954,169	56.2
Etiocholenolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	12,280,836	78.7
5a Androstan 3a 17b diol diAC	256	2,634,418	50.5
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	4,508.073	27.2

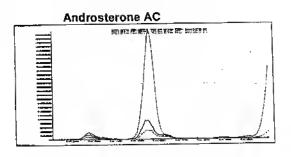
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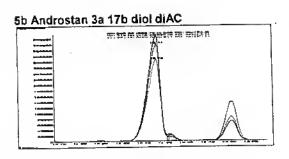
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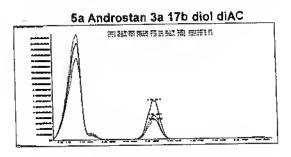
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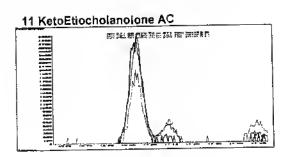


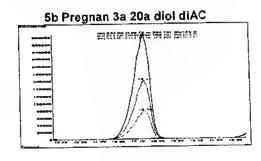












Page 2 of 2

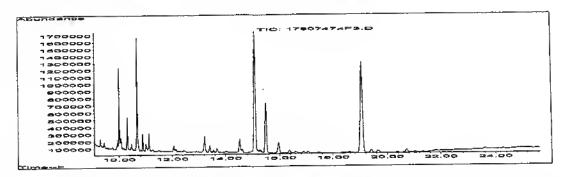
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Data File Name 17807474F3.D Data File Path D:\Msd22\Juil06\2307\ Operator 49 Date Acquired 7/23/2006 11:33 Acq. Method File MAN_52.M Sample Name 178/07 995474 F3

Vial Number 3 Misc Info 178/07 995474 Fraction 3 dans 400µL



Temps de rétention, temps de rétention relatif et target Signal (M1)

Name	Ret Time	Rel Ret Time	Target Signal	Target Response
5a Androstanol AC	10.67		258	2,492,729
Etiocholanolone AC	0.00	0.000	272	0
Androsterone AC	0.00	0.000	272	0
5b Androstan 3a 17b diol diAC	15.10	1.415	256	3,658,105
5a Androstan 3a 17b diol diAC	15.48	1.450	316	1,968,201
11 KetoEtiocholanolone AC	0.00	0,000	271	0
5b Pregnan 3a 20a diol diAC	19.06	1.786	284	4,951,688

M2 signal

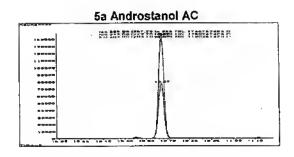
Name	Q1 signal	Q1 Response	Q1 Ratio
5a Androstanol AC	243	2,615,044	104.9
Etiochotanolone AC	257	0	0.0
Androsterone AC	257	0	0.0
5b Androstan 3a 17b diol diAC	316	3,242,469	88.6
5a Androstan 3a 17b diol diAC	241	1,199,926	61.0
11 KetoEtiocholanolone AC	191	0	0.0
5b Pregnan 3a 20a diol diAC	269	2.833.947	57.2

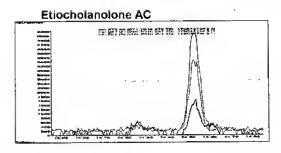
M3 signal

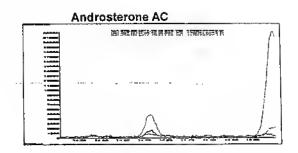
	O2 signal	Q2 Response	Q2 Ratio
Name	Q2 signal		
5a Androstanol AC	204	1,395,444	56.0
Etiocholanolone AC	201	0	0.0
Androsterone AC	218	0	0.0
5b Androstan 3a 17b diol diAC	241	2,877,543	78.7
5a Androstan 3a 17b diol diAC	256	985,712	50.1
11 KetoEtiocholanolone AC	286	0	0.0
5b Pregnan 3a 20a diol diAC	344	1,343,347	27.1

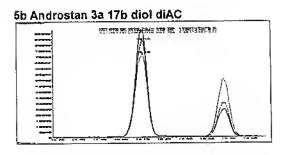
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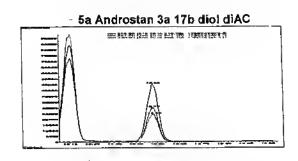
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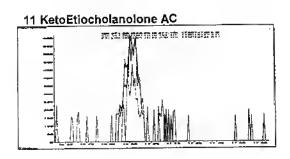


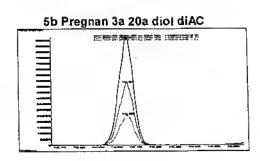












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7/23/2006 12:00 PM

LNDD

ENREGISTREMENT

Codification: E-CC-11

Version: B

Date: 08/03/2006

1/1

VERIFICATION DES PERFORMANCES INSTRUMENTALES EN CG/SM (screening et confirmation)

Numé	ro d'identification de l'ap	pareil : المنافقة متاكا	
		Date: 13/07/06	
1 - Source d'	<u>ionisatioo</u>		Oui Non
MSD	Autotune: Ion 69 ou 2 Autotune: Abondance Repeller < 35		× ×
Polaris	Ion time > 2 ms		
Observations	: .		
2 - Etanchéi	té do système	*:	Oui Non
MSD	18/69 (H2O), 28/69 (44/69 (CO2) < 10%	N2), 32/69 (O2),	A
Polaris	. ,	on 19 < intensité ion I	8
Observations	: :		
3 - Sensihili	<u>tě</u>	·	Oui Non
Screening	Recal / Mix conform	c	
Conf	TP conforme - Fichi		
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Observation	s:		
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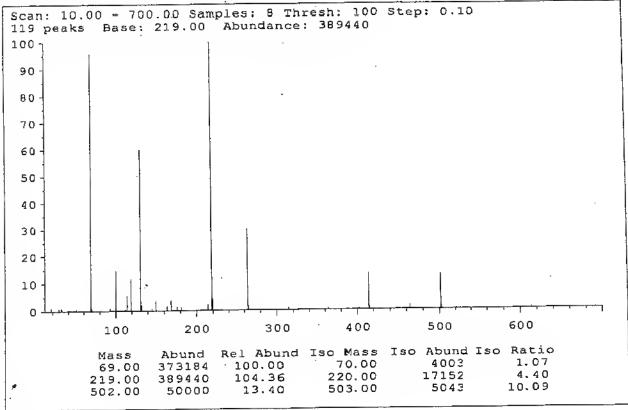
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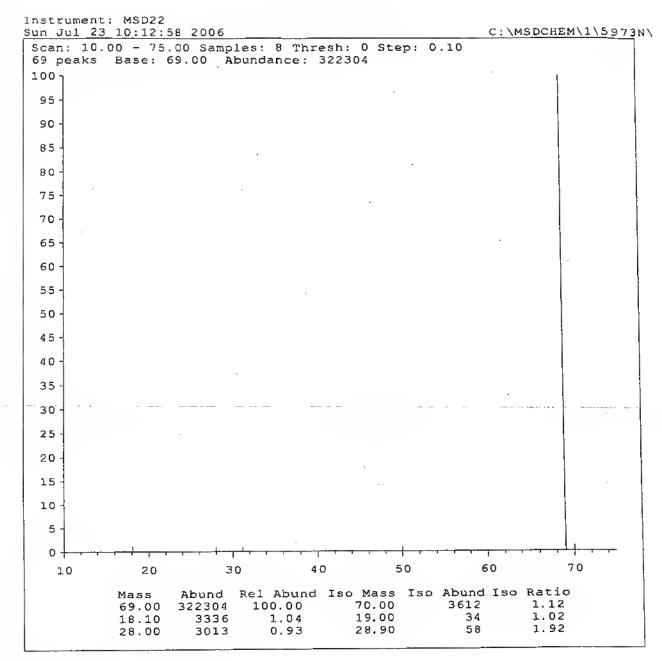
Cet enregistrement est à archiver dans le classeur C-MA-Ech de l'appareil

sun Jul 23 10:11:55 2006
C:\MSDCHEM\1\5973N\ATUNE.U

Instrument: MSD22

Ab 421715	Mass	69.00	Mass	219.00	Mass	502.00	Ton Pol POS MassGain 267
Pw50 0.61 Pw50 0.61 Pw50 0.62 Emission 34.6 AmuGain 2179 ElEnrgy 69.9 AmuOffs 130 DC Pol NEO				437153	Ab	54028	
Filament 2 Wid219 -0.037 DC Pol NEC Repeller 24.26 IonFcus 90.2 HEDEnab ON EntLens 21.0 EMVolts 1812 EntOffs 17.57 Samples PFTBA OPEN Averages Stepsize 0.10 Zones: MS Source 230 Foreline 9	Pw50	0.61	Pw50	, 0.61	PW5U	, 0.62	
Repeller 24.26 IonFcus 90.2 HEDEnab On EntLens 21.0 EMVolts 1812 EntOffs 17.57 Samples 8 PFTBA OPEN Averages Stepsize 0.16 Zones: MS Source 230 Foreline 9	ļ	1		A 1		A 1	
Repeller 24.26 IonFcus 90.2 HEDEnab OtentLens 21.0 EMVolts 1812 EntOffs 17.57 Samples 8 PFTBA OPEN Averages Stepsize 0.16 Zones: MS Source 230 Foreline 9	1			1) 1			
IonFcus 90.2 HEDEnab On EntLens 21.0 EMVolts 1812 EntOffs 17.57 Samples 8 PFTBA OPEN Averages Stepsize 0.16 Zones: MS Source 230 Foreline 9						11	
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Samples 8 PFTBA OPEN Averages Stepsize 0.1 Zones: MS Source 230 Foreline 9							
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Current Params used: ATUNE.U

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Relative abundances:

18/69 = 1.04 Water%

28/69 = 0.93 Nitrogen%

32/69 = 0.28 Oxygen%

44/69 = 0.17 Carbon Dioxide%

28/18 = 90.32 Nitrogen/Water%
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Echantillon:		178/07 9	95474							
Folérances fixées p)			
Coloranoes sur no u	ot it ar , in	-1 70 OU 17-	o,z mm (pr	entere la pro	is tallyte de.	s deux)				
Pour les abondance		> 50% 25 <ab< 50<br="">< 25%</ab<>		admis	+/- 10% (6 +/- 20% (6 +/- 5% (er	•	e variation			
Calcul des abondan	ices en :	1	Γabυłation:			Surface:	х		Hauteur:	
Standard Interne:			·	"						
	М	ix	Fracti	on F1	Fract	ion F2	Fracti	on F3		
Tr (min)		.69	10.			.69	10.			
Fichier	2307M	ixAc01	17807	474F1	17807	474f2b	178074	474F3		
Substance carac	rtérisée · Í	11 Kétos	tiocholanol	Ione AC		Fichier ·	1	78074 7 4F1		-
Substance carac	, 		Mix		147 (0/)	Fichier : [F	7807474F1	M2 (9/2)	3.52 (0/)
Substance carac	Tr (min)	Trr	Mix M1 (%)	M2 (%)	M3 (%)	Tr (min)	F Trr	chantillon M1 (%)	M2 (%)	M3 (%)
	Tr (min) 17.07	Trr 1.597	Mix	M2 (%)	59.9		F	chantillon	M2 (%) 81.1	M3 (%) 60.7
Tolérance basse	Tr (min) 17.07 16.90	Trr 1.597 : 1.581	Mix M1 (%)	M2 (%)		Tr (min)	F Trr	chantillon M1 (%)		
Tolérance basse Tolérance haute	Tr (min) 17.07 16.90 17.24	Trr 1.597	Mix M1 (%)	M2 (%) 83 73	59.9 49.9 69.9	Tr (min)	F Trr	chantillon M1 (%)		
Tolérance basse Tolérance haute Concordance des T	Tr (min) 17.07 16.90 17.24	Trr 1.597 : 1.581	Mix M1 (%)	M2 (%) 83 73 93	59.9 49.9 69.9	Tr (min) 17.05	F Trr	chantillon M1 (%)		
Tolérance basse Tolérance haute Concordance des T Concordance des T	Тr (min) 17.07 16.90 17.24	Trr 1.597 1.581 1.613	Mix M1 (%) 100	M2 (%) 83 73 93 oni oui	59.9 49.9 69.9 X	Tr (min) 17.05 non non	F Trr	chantillon M1 (%)		
Tolérance basse Tolérance haute Concordance des T	Тr (min) 17.07 16.90 17.24	Trr 1.597 1.581 1.613	Mix M1 (%) 100	M2 (%) 83 73 93	59.9 49.9 69.9	Tr (min) 17.05	F Trr	chantillon M1 (%)		
Tolérance basse Tolérance haute Concordance des T Concordance des T	Tr (min) 17.07 16.90 17.24 r: r: le des abond	Trr 1.597 1.581 1.613	Mix M1 (%) 100	M2 (%) 83 73 93 oni oui oui	59.9 49.9 69.9 X	Tr (min) 17.05 non non	Trr 1.596	chantillon M1 (%)	81.1	
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa	Tr (min) 17.07 16.90 17.24 r: r: le des abond	Trr 1.597 1.581 1.613	Mix M1 (%) 100 ves:	M2 (%) 83 73 93 oni oui oui	59.9 49.9 69.9 X	Tr (min) 17.05 non non non	Trr 1.596	M1 (%)	81.1	
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa	Tr (min) 17.07 16.90 17.24 r: r: le des abond	Trr 1.597 1.581 1.613 ances relati	Mix M1 (%) 100	M2 (%) 83 73 93 oni oui oui	59.9 49.9 69.9 X	Tr (min) 17.05 non non non	Trr 1.596	7807474F2	81.1	60.7
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa	Tr (min) 17.07 16.90 17.24 r: r: le des abond	Trr 1.597 1.581 1.613	Mix M1 (%) 100 ves:	M2 (%) 83 73 93 oni oui oui	59.9 49.9 69.9 X X X	Tr (min) 17.05 non non non Fichier:	1.596	Chantillon M1 (%) 100 7807474F2 Echantillon	81.1 b	60.7
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa	Tr (min) 17.07 16.90 17.24 r: r: le des abond ctérisée :	Trr 1.597 1.581 1.613 ances relati	Mix M1 (%) 100 ves : cholanolons Mix M1 (%)	M2 (%) 83 73 93 oui oui oui	59.9 49.9 69.9 X X X X 42.6 34.08	Tr (min) 17.05 non non non Tr (min)	1 Tπ	7807474F2 Echantillon M1 (%)	b M2 (%)	60.7 M3 (%)
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa Substance carac	Tr (min) 17.07 16.90 17.24 r: r: de des abond etérisée : Tr (min) 14.35	Trr 1.597 1.581 1.613 lances relati Etio Trr 1.343	Mix M1 (%) 100 ves : cholanolons Mix M1 (%)	M2 (%) 83 73 93 oni oui oui oui 63.8	59.9 49.9 69.9 X X X X 42.6	Tr (min) 17.05 non non non Tr (min)	1 Tπ	7807474F2 Echantillon M1 (%)	b M2 (%)	60.7 M3 (%)
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa Substance carac Tolérance basse Tolérance haute	Tr (min) 17.07 16.90 17.24 r: r: le des abond etérisée : Tr (min) 14.35 14.21 14.49	Trr 1.597 1.581 1.613 lances relation Etio Trr 1.343 1.330	Mix M1 (%) 100 ves : cholanolons Mix M1 (%)	M2 (%) 83 73 93 oni oui oui oni AC M2 (%) 63.8 53.8 73.8	59.9 49.9 69.9 X X X X X 34.08 51.12	Tr (min) 17.05 non non non Tr (min) 14.38	1 Tπ	7807474F2 Echantillon M1 (%)	b M2 (%)	60.7 M3 (%)
Tolérance basse Tolérance haute Concordance des T Concordance des T Concordance globa Substance carac	Tr (min) 17.07 16.90 17.24 r: r: le des abond etérisée : Tr (min) 14.35 14.21 14.49	Trr 1.597 1.581 1.613 lances relation Etio Trr 1.343 1.330	Mix M1 (%) 100 ves : cholanolons Mix M1 (%)	M2 (%) 83 73 93 oui oui oui oui 63.8 53.8	59.9 49.9 69.9 X X X X 42.6 34.08	Tr (min) 17.05 non non non Tr (min)	1 Tπ	7807474F2 Echantillon M1 (%)	b M2 (%)	60.7 M3 (%)

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ENREGISTREMENT

Codification:

E-FCR-09

Version: Date:

31/01/2006

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FICHE D'ANALYSE / RESULTAT ANALYSE QUALITATIVE GC/MS POUR CONFIRMATION GC/C/IRMS

Substance caractérisée : [And	rostérone A	C		Fichier:	17	17807474F2b			
1	<u> </u>		Mix		<u></u>		E	Echantillon			
	Tr (min)	Trr	Mi (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%	
	14.62	1.367	100	42.6	30.6	14.65	1.370	100	43	30.2	
Tolérance basse	14.47	1.353	100	34.08	24.48					-	
Tolérance haute	14.77	1.381		51.12	36.72						
Tolerance Baute	14177	1.501				_		_			
Concordance des Ti	r:			oui	X	поп					
Concordance des Ti	m:			oui	X	non		ļ			
Concordance globa	le des abonda	ances relati	ves:	oui	X	поп [j 			
Substance carac	ctérisée :	5b Andros	tane-3a,17b-	diol diAC		Fichier: [1	7807474F	3]	
			Mix	· · · · · · · · ·			I	chantillon			
	Tr (min)	Тп	M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%	
	15.17	1.419	100	90.4	. 77.1	15.1	1.415	100	88.6	78.7	
Tolérance basse	15.02	1.405		80.4	67.1			-			
- Tolérance haute	15-32	1.433		100.4	87.1 -						
Concordance des T				oui	X	non] .			
				oui	$\overline{\mathbf{x}}$	non		1			
Concordance des T	m:			Out	1 48						
Concordance des T Concordance globa		iances relat	ives :	oui	<u>x</u>	non] 	•••		
	ale des abond		ives : stane-3a,17b	oui		4		178074741	73]	
Concordance globa	ale des abond		stane-3a,17b	oui		non		17807474I]	
Concordance globa	ale des abond ctérisée :	5a Andro	stane-3a,17b Mix	oui -diol diAC]	non Fichier:			1] M3 (
Concordance globa	ale des abond ctérisée : Tr (min)	5a Andro	stane-3a,17b Mix M1 (%)	oui -diol diAC - M2 (%)	X	non		Echantillo	1		
Concordance globa Substance cara	ctérisée : Tr (min) 15.57	5a Andre Trr 1.456	stane-3a,17b Mix	oui -diol diAC -diol diAC -diol 58.5	X M3 (%) 48.9	Fichier:	Trr	Echantillor	n M2 (%)		
Concordance globa Substance cara Tolérance basse	ctérisée : Tr (min) 15.57 15.41	5a Andro Trr 1.456 1.441	stane-3a,17b Mix M1 (%)	oui -diol diAC M2 (%) 58.5 48.5	X	Fichier:	Trr	Echantillor	n M2 (%)		
Concordance globa Substance cara	ctérisée : Tr (min) 15.57	5a Andre Trr 1.456	stane-3a,17b Mix M1 (%)	oui -diol diAC -diol diAC -diol 58.5	M3 (%) 48.9 39.12	Fichier:	Trr	Echantillor	n M2 (%)		
Substance cara Tolérance basse Tolérance haute	Tr (min) 15.57 15.41 15.73	5a Andro Trr 1.456 1.441	stane-3a,17b Mix M1 (%)	oui -diol diAC M2 (%) 58.5 48.5	M3 (%) 48.9 39.12	Fichier:	Trr	Echantillor	n M2 (%)		
Substance cara Tolérance basse Tolérance haute Concordance des T	r:	5a Andro Trr 1.456 1.441	stane-3a,17b Mix M1 (%)	oui -diol diAC -diol diAC -58.5 -48.5 -68.5	X M3 (%) 48.9 39.12 58.68 X X	Fichier: Tr (min) 15.48	Trr	Echantillor	n M2 (%)		
Substance cara Tolérance basse Tolérance haute Concordance des Toncordance d	Tr (min) 15.57 15.41 15.73	5a Andro Trr 1.456 1.441 1.471	Mix M1 (%) 100	oui -diol diAC M2 (%) 58.5 48.5 68.5	X M3 (%) 48.9 39.12 58.68	Fichier: Tr (min) 15.48	Trr	Echantillor	n M2 (%)		
Substance cara Tolérance basse Tolérance haute Concordance des T	Tr (min) 15.57 15.41 15.73	5a Andro Trr 1.456 1.441 1.471	Mix M1 (%) 100	oui -diol diAC -diol d	X M3 (%) 48.9 39.12 58.68 X X	Fichier: Tr (min) 15.48	Trr	Echantillor	n M2 (%)		
Substance cara Tolérance basse Tolérance haute Concordance des Toncordance d	ctérisée : Tr (min) 15.57 15.41 15.73 Tr : Tr : ale des abone	5a Andro Trr 1.456 1.441 1.471 dances rela	Mix M1 (%) 100	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui	X M3 (%) 48.9 39.12 58.68 X X	Fichier: Tr (min) 15.48	Trr	Echantillor	M2 (%)		
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance des Tolerance glob	ctérisée : Tr (min) 15.57 15.41 15.73 Tr : Tr : ale des abone	5a Andro Trr 1.456 1.441 1.471 dances rela	Mix M1 (%) 100 tives:	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui	X M3 (%) 48.9 39.12 58.68 X X	Fichier: Tr (min) 15.48 non non	Trr	Echantillo M1 (%) 100 17807474 Echantillo	F3	50.	
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance des Tolerance glob	ctérisée : Tr (min) 15,57 15,41 15,73 Tr : Tr : ale des abond	5a Andro Trr 1.456 1.441 1.471 dances rela	Mix M1 (%) 100	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui oui	X M3 (%) 48.9 39.12 58.68 X X	Fichier: Tr (min) 15.48 non non ron	Тп i.450	Echantillo M1 (%) 100 17807474 Echantillo M1 (%)	F3 M2 (%)	50.	
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance des Tolerance glob	ctérisée : Tr (min) 15.57 15.41 15.73 Tr : ale des abond actérisée : Tr (min)	5a Andro Trr 1.456 1.441 1.471 dances rela	Mix M1 (%) 100 tives:	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui oui	X M3 (%) 48.9 39.12 58.68 X X	Fichier: Tr (min) 15.48 non non ron	1.450	Echantillo M1 (%) 100 17807474 Echantillo	F3	50.	
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance des Tolerance glob Substance cara	ctérisée : Tr (min) 15.57 15.41 15.73 Tr : ale des abone actérisée : Tr (min) 19.2	5a Andro Trr 1.456 1.441 1.471 dances rela 5b Prég	Mix M1 (%) 100 tives:	oui diol diAC M2 (%) 58.5 48.5 68.5 oui oui oui oui diol diAC	X M3 (%) 48.9 39.12 58.68 X X X	Fichier: Tr (min) 15.48 non non non Tr (min)	Тп i.450	Echantillo M1 (%) 100 17807474 Echantillo M1 (%)	F3 M2 (%)	50.	
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance glob Substance cara	ctérisée : Tr (min) 15.57 15.41 15.73 Tr : Tr : ale des abone actérisée : Tr (min) 19.2 19.01	5a Andro Trr 1.456 1.441 1.471 dances rela Sb Prég Trr 1.796 1.778	Mix M1 (%) 100 tives:	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui oui -diol diAC	X M3 (%) 48.9 39.12 58.68 X X X 28	Fichier: Tr (min) 15.48 non non non Tr (min)	Тп i.450	Echantillo M1 (%) 100 17807474 Echantillo M1 (%)	F3 M2 (%)	50.	
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance des Tolerance glob Substance cara	ctérisée : Tr (min) 15.57 15.41 15.73 Tr : Tr : ale des abone actérisée : Tr (min) 19.2 19.01	5a Andro Trr 1.456 1.441 1.471 dances rela 5b Prég	Mix M1 (%) 100 tives:	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui oui -diol diAC	X M3 (%) 48.9 39.12 58.68 X X X 28 22.4	Fichier: Tr (min) 15.48 non non non Tr (min)	Тп i.450	Echantillo M1 (%) 100 17807474 Echantillo M1 (%)	F3 M2 (%)	50 M3	
Substance cara Tolérance basse Tolérance haute Concordance des Tolerance glob Substance cara	Tr (min) 15.57 15.41 15.73 Tr : Trr : ale des abone actérisée : Tr (min) 19.2 19.01 19.39	5a Andro Trr 1.456 1.441 1.471 dances rela Sb Prég Trr 1.796 1.778	Mix M1 (%) 100 tives:	oui -diol diAC M2 (%) 58.5 48.5 68.5 oui oui oui -diol diAC	X M3 (%) 48.9 39.12 58.68 X X X 28 22.4	Fichier: Tr (min) 15.48 non non non Tr (min)	Тп i.450	Echantillo M1 (%) 100 17807474 Echantillo M1 (%)	F3 M2 (%)	50 50 M3	

oui

Concordance globale des abondances relatives :

non

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FICHE D'AN	NALYSE / R	ESULTA1	T ANALYS	E QUALI	TATIVE	GC/MS PO	UR CONF	IRMATIO	N GC/C/II	RMS_
	_									
Substance carac	térisée :					Fichier:				
	<u> </u>		Mix		Echantillon					
	Tr (min)	Тπ	Ml (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (%
			100					100		
Tolérance basse										
Tolérance haute	<u> </u>		:							
Concordance des T	۲.			oui		non		3		
Concordance des T				oui		non		†		
Concordance globa		ınces relati	ives:	oni	 -	non		1		
Substance carac	térisée :]	Fichier:]
) C.,		-			Echantillon		
	Tr (min)	Тп	Mix M1 (%)	M2 (%)	M3 (%)	Tr (min)	Trr	M1 (%)	M2 (%)	M3 (9
recommended to the re-	11 (ium)	111	100	1912 (70)	1415 (70)	11 (11111)		100	1712 (70)	1115 (
Tolérance basse								-		
Tolérance haute			1							"
Concordance des T	r:			oui		non		7		
Concordance des T				oni		non		7		
Concordance globa		ances relat	ives:	oui		non]		
		,								
Paraphe et code op	érateur :		49 1	ang						
Partie à remplir par	le responsab	le :								
Caractérisation for	melle de tous	les analyte	es:	оці	X	non				
Paraphe et code op	érateur :		10	CB						
Observations:									•	
	Cet enregisi	trement es	t å archiver	dans le do	ossier de co	onfirmation	de l'échanti	illon		